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TOTAL GAMMA COUNT RATE ANALYSIS METHOD FOR NONDESTRUCTIVE ASSAY CHARACTERIZATION

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Abstract

A new approach to nondestructively characterize waste for disposal, based on total gamma response, has been developed at the Idaho Cleanup Project by CH2M-WG Idaho, LLC and Idaho State University, and is called the total gamma count rate analysis method. The total gamma count rate analysis method measures gamma interactions that produce energetic electrons or positrons in a detector. Based on previous experience with waste assays, the radionuclide content of the waste container is then determined. This approach potentially can yield minimum detection limits of less than 10 nCi/g.

The importance of this method is twofold. First, determination of transuranic activity can be made for waste containers that are below the traditional minimum detection limits. Second, waste above 10 nCi/g and below 100 nCi/g can be identified, and a potential path for disposal resolved.

Introduction

A new approach to nondestructively characterize waste for disposal has been developed at the Idaho Cleanup Project (ICP) as a collaborative project between CH2M-WG Idaho, LLC and Idaho State University. This method is based on total gamma response and is called the total gamma count rate (TGCR) analysis method. The method may be used to characterize and "load manage" waste slated for disposal at the Waste Isolation Pilot Plant (WIPP), or dispose of waste at an alternate disposal facility. In most circumstances, the total gamma count rate analysis can lower the detection limit of a gamma spectrometer system to or below 10 nCi/g.

Waste generated by atomic energy defense-related activities with a concentration between 10 and 100 nCi/g is often called "orphan" waste because currently there is no facility where this waste can be stored without treatment. Load management allows the U.S. Department of Energy (DOE) to send atomic energy defense-related waste for disposal at WIPP. Alternately, if the waste is characterized below 10 nCi/g, the waste may be sent to a Class C disposal facility. Disposal at a Class C facility is significantly less expensive than disposal at WIPP. Nondestructive characterizing of "orphan" waste is not only important to ICP but DOE, in general. The benefit to ICP and DOE facilities will be that waste once unavailable for disposition can now be disposed of, thereby, reducing the legacy waste around the DOE complex. The ICP and the Advanced Mixed Waste Treatment Facility are aggressively pursing this technique to characterize waste for disposal.

The TGCR analysis method measures the sum of gamma interactions that occur; these interactions consist of the photoelectric effect, Compton scattering, and electron-positron pair production. The total count rate is proportional to the gamma-emitting activity in the sample, and, assuming a strong correlation exists between gamma activity and transuranic activity, the total count rate can be used to determine the amount of transuranic material present.

Normal passive gamma-ray spectrometry is based on interactions where all the energy of the photon is deposited in the detector, while the TGCR method is based on all gamma interactions that occur within the detector. In addition, the TGCR method is sensitive to multiple gamma-ray scattering that not only occurs in the detector, but in the waste matrix in a low-background environment. As a result, the TGCR method is more sensitive to total gamma activity than normal gamma spectrometry. However, the TGCR method's sensitivity does rely on having a low background

or a background that is highly controlled. The TGCR method is similar to neutron counting in that neither technique can provide isotopic data by itself. Therefore, in order to convert gamma response (or neutron response) to transuranic activity, relative isotopic information must be known about the waste. This information can be obtained through measurement experience for each waste category where isotopic data are determined or by process knowledge on how the waste was generated.

Total Gamma Count Rate

The TGCR is a measure of gamma activity originating in waste. The theory is that TGCR, as measured by a simple gamma-detector or integrated over the energy spectrum of a gamma spectrometer, is proportional to the gamma-emitting activity in the sample. Assuming that a strong correlation exists between gamma activity and transuranic activity, TGCR can be used to determine the amount of transuranic material present in a waste container.

Example scenarios where a photon emitted in the matrix leads to a count in the detector are shown in Figure 1. Once a photon interacts within a detector, there are two possibilities that can occur: 1) all energy from the photon is deposited, 2) the photon is partially absorbed and the remaining photon's energy escapes the detector[1]. With normal gamma spectroscopy, only energy that is fully deposited in the detector (i.e., under photopeaks) is used to determine the activity in a sample/container; however, TGCR accounts for all energy of a photon whether or not it is fully deposited in the detector. The drawback of TGCR is that isotopic ratios must be known about the waste. In the case of Idaho National Laboratory plutonium, isotopic mass ratios are well documented and are shown in Table 1.

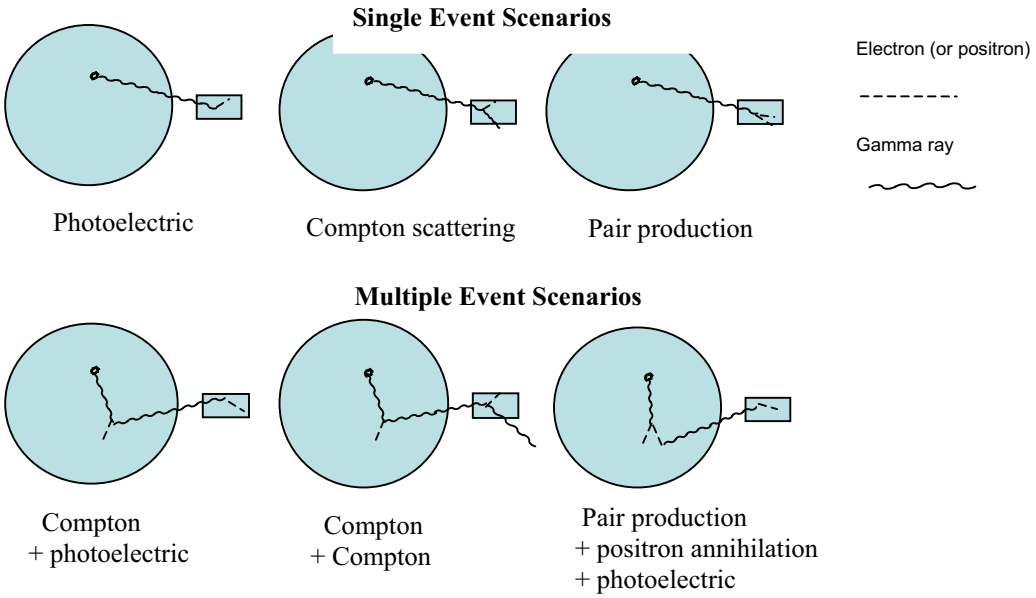

Figure 1. Example scenarios where a gamma ray emitted in the matrix can lead to a count in the detector.

Table 1. Default weapons-grade plutonium isotopic mass ratio of waste generated from the Rocky Flats Plant and stored at the Idaho National Laboratory[2].

Isotope	Default Plutonium Mass Fraction[a]	Standard Deviation
Pu-238	1.05E-4	4.1E-5
Pu-239	9.406E-1	4.9E-3
Pu-240	5.72E-2	4.8E-3
Pu-241	1.73E-3	3.2E-4
Pu-242	4.3E-4	2.2E-4

a. These are default plutonium mass fractions of the activity present in the waste.

There are three phases of work that have or will be accomplished: 1) a review of the database for the following waste categories at the Idaho National Laboratory Site—filter waste, inorganic (or aqueous) sludge waste, organic sludge waste, and graphite molds (referred to as the parametric study); 2) surrogate waste measurements using a typical high-purity germanium detector and surrogate drums of interest; and 3) Monte Carlo N-Particle calculations.

Parametric Study

This parametric study is an in depth study of gamma-ray spectrometer data collected during the 3,100 m^3 Project at the Idaho National Laboratory Site from 1997 through 2002. The project involved characterization and shipment of 3,100 m^3 of transuranic waste to WIPP. The relative isotopic concentrations and waste form characteristics of Rocky Flats Plant waste, as shown in Table 1, are assumed to be representative of waste to be retrieved during excavation of pits at the Idaho National Laboratory Site, and will be used as the basis for ensuing parametric studies.

The data set used to perform this work was comprised of 3,500 assay files that were used to evaluate the TGCR under realistic assay conditions. A random subpopulation of 321 assay records was used to calibrate the TGCR method. Then the remainder of the assay records was compared against the calibrated TGCR results. This data set is comprised of waste containers that were

1. Assayed using only the Stored Waste Examination Pilot Plant gamma-ray system to ensure that waste assay configuration was always the same
2. All data sets were reanalyzed with the same version gamma spectrum analysis code to ensure results were comparable (This was made necessary due to that over the history of the 3,100 m^3 operations, the Stored Waste Examination Pilot Plant gamma-ray system had analyzed gamma-ray spectra using different versions of assay software. Reanalyzing the data using the same version of the gamma spectrum analysis code was made possible because the format of the basic spectrometer data had remained the same over this period.)
3. Contained targeted waste identified for retrieval from pits.
    a. Aqueous sludge, IDCs 1 and 2. Subpopulation is 100 waste assays.
    b. Organic sludge, IDC 3. Subpopulation is 21 waste assays.
    c. Graphite, IDCs 300, 303, 310, 311, and 312. Subpopulation is 100 waste assays.
    d. Filters, IDCs 335, 376, and 490. Subpopulation is 100 waste assays.

Measured Versus Calculated Response

The measured gamma response was the total count rate for the spectrum of interest and is called GR_m. The calculated gamma response (GR_{calc}) is shown in Equation 1. The measured versus the calculated gamma response was determined to be a linear function for all waste types of interest.

Equation 1
$$GR = \langle eff \rangle \times \sum_i M_i \, SA_i \sum_g b_{ig} \, \varepsilon_{tot}\left(E_{ig}\right) = \langle eff \rangle \times GA$$

Where:

GR is the gamma response (or total gamma count rate) in units of counts per second (cps)

eff is the matrix specific calibration coefficient determined from comparisons of GR_{calc} with GR_{meas}

M_i is the mass of isotope "*i*" in units of grams (g)

SA_i is the specific activity of isotope "*i*" in units of Bq/g

b_{ig} is the fractional gamma branching ratio for gamma line "*g*" from the decay of isotope "*i*"

$\varepsilon_{tot}(E_{ig})$ is the total efficiency of the detector response due to an emitted gamma-ray at energy E_{ig}

E_{ig} is the energy of the gamma line "*g*" from the decay of isotope "*i*"

GA is the gamma activity.

The matrix dependent calibration coefficient, $\langle eff \rangle$, is determined by linear fit to the data presented in Figure 2 and was determined to be 0.1664. Additional matrix dependent calibration coefficients are shown in Table 2. Based on Equation (1), the linear fit is forced to have a "0" intercept.

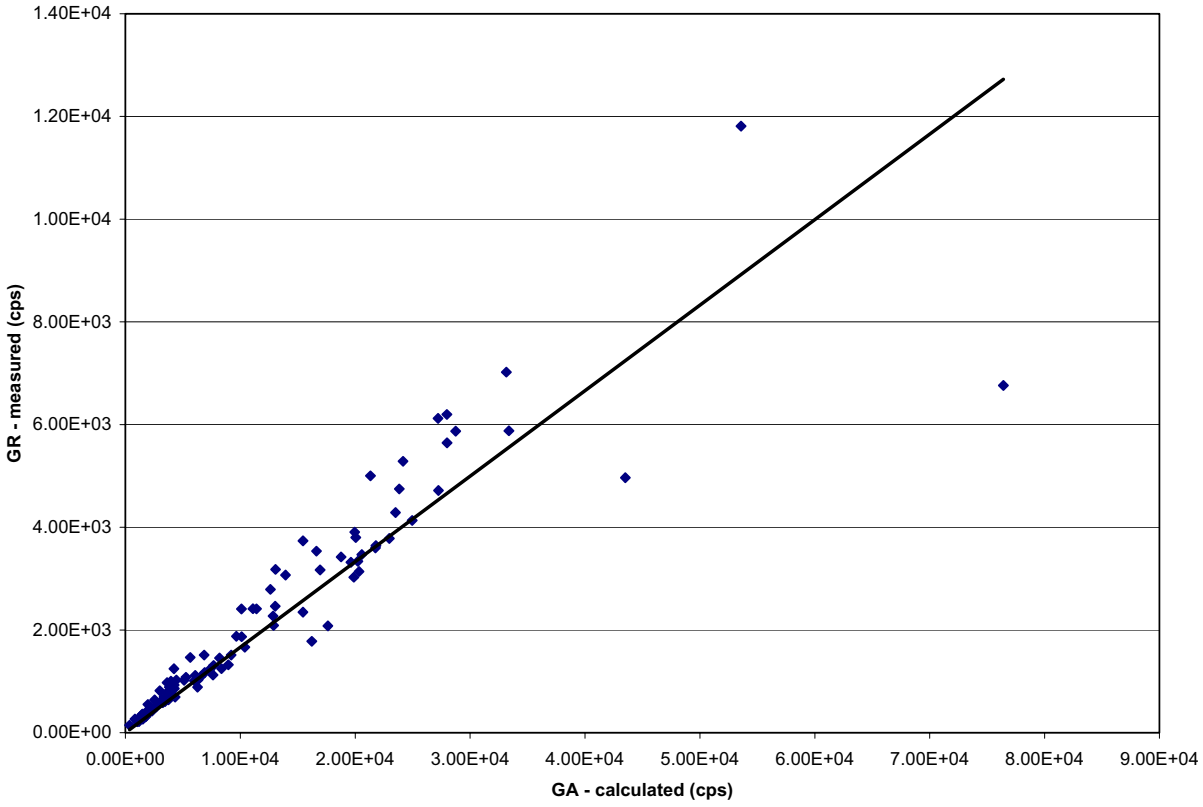

Figure 2. Calculated versus measured total count rate for aqueous sludge.

Table 2. Matrix gamma attenuation coefficients.

Matrix	$\langle eff \rangle$
Aqueous sludge	0.1664

Organic sludge	0.3001
Graphite	0.3969
Filter	0.5744

The correlation demonstrated here for aqueous sludge is the worst case scenario, and the correlations for other waste categories were much better. While it is expected that the calibration factor would be waste-content code-specific, it was found that it may be possible to combine the TGCR calibration for all waste types, except for aqueous sludge, without a significant loss in accuracy. This concept will be described at later time.

Relative Isotope Contribution to Gamma Activity

The database can also be used to arrive at the mean relative concentration and uncertainty for each isotope, *"k"*. The relative contribution of an isotope "*k*" to the total gamma count rate is defined using Equation 2:

Equation 2
$$R_k \equiv \frac{M_k \; SA_k \sum_g b_{kg} \; \varepsilon_{tot}\left(E_{kg}\right)}{GA}$$

Where:

R_k is the relative gamma response contribution from isotope "*k*" (all other parameters are defined in Equation 1).

Using the subset population of previous waste assays, relative gamma contributions for the respective isotopes can be extracted, and from the data set of R_k*'s*, the mean, $\langle R_k \rangle$, and its standard deviation, σ_{R_k}, can be determined for each "*k*" isotope. The relative contribution of each WIPP isotope that can be measured with a gamma spectrometer is shown in Table 3. To the right of each isotope's R_k value is the ranking of that isotope's contribution to the total gamma response for that waste category. As expected, the two top contributors to the gamma response are Pu-239 and Am-241. These two isotopes contribute approximately 94% of the total gamma response. However, the rankings of these two isotopes change between aqueous sludge and the remainder of the waste categories. In aqueous sludge, Am-241 ranks first and Pu-239 second; however, for all other waste categories Pu-239 ranks first and Am-241 ranks second.

Table 3. Relative gamma contribution for each isotope of interest.

	Aqueous Sludge $\langle R_k \rangle$ [a]	Organic Sludge $\langle R_k \rangle$ [a]	Graphite $\langle R_k \rangle$ [a]	Filters $\langle R_k \rangle$ [a]
Rk_Pu238	(3.44 ± 4.1)E-04[7]	(2.55 ± .54)E-03[5]	(2.67 ± .11)E-03[5]	(2.58 ± .73)E-03[6]
Rk_Pu239	(9.39 ± 11.)E-02[2]	(6.77 ± 1.4)E-01[1]	(7.40 ± .31)E-01[1]	(7.09 ± 2.0)E-01[1]
Rk_Pu240	(2.29 ± 2.8)E-03[6]	(1.70 ± .36)E-02[4]	(1.77 ± .08)E-02[4]	(1.72 ± .48)E-02[4]
Rk_Pu241	(5.37 ± 6.5)E-03[5]	(3.97 ± .84)E-02[3]	(4.15 ± .18)E-02[3]	(4.03 ± 1.1)E-02[3]
Rk_Pu242[b]	(0.00 ± .00)E+00	(0.00 ± .00)E+00	(0.00 ± .00)E+00	(0.00 ± .00)E+00
Rk_Am241	(8.51 ± 1.6)E-01[1]	(2.57 ± 1.5)E-01[2]	(1.98 ± .34)E-01[2]	(2.32 ± 2.2)E-01[2]
Rk_U233[b]	(-2.43 ± 6.1)E-04	(-0.52 ± 74.)E-04	(0.66 ± 13.)E-04	(-4.87 ± 49.)E-03
Rk_U234	(1.69 ± 3.1)E-05[8]	(2.85 ± 6.0)E-06[8]	(1.10 ± 21.)E-08[8]	(5.38 ± 37.)E-06[8]
Rk_U235	(1.30 ± 2.4)E-02[4]	(2.12 ± 4.4)E-03[7]	(0.91 ± 18.)E-05[6]	(4.19 ± 29.)E-03[5]
Rk_U238	(3.43 ± 7.4)E-02[3]	(4.41 ± 12.)E-03[6]	(0.08 ± 19.)E-05[7]	(9.64 ± 83.)E-05[7]
Rk_CS137[b]	(0.00 ±.00)E+00	(0.00 ± .00)E+00	(0.00 ± .00)E+00	(0.00 ± .00)E+00

a. The number, X, given in [X] is the ranking of the isotope's contribution to the total gamma response.
b. These isotopes were not ranked.

Isotope Mass Calculation

The application of TGCR to the Contact-Handled Waste Acceptance Criteria requirements entails breaking down the measured total count rate into its isotopic components. Then the "derived parameters" must be calculated from the isotopic components. The isotope "*k*" gamma activity from isotope "*k*" mass can be determined in Equation 3.

Equation 3

$$\left\langle GA_m \right\rangle = \frac{GR_m}{\left\langle eff \right\rangle}$$

$$\left\langle GA_k^m \right\rangle = \left\langle R_k \right\rangle \left\langle GA_m \right\rangle$$

$$\left\langle M_k^m \right\rangle = \frac{\left\langle GA_k^m \right\rangle}{SA_k \sum_g b_{kg}\, \varepsilon_{tot}\left(E_{kg}\right)}$$

Where:

The subscript (or superscript) *m* refers to the measured (or determined from measurement) value

GR_m is the measured total count rate

$\left\langle GA_m \right\rangle$ is the estimated gamma activity, determined by dividing the measured total count rate by the matrix specific calibration factor

$\left\langle GA_k^m \right\rangle$ is the estimated gamma activity due to "k^{th}" isotope, based on the total count rate method

$\left\langle M_k^m \right\rangle$ is the estimated mass of the isotope "*k.*"

Figure 3 shows the estimated isotope mass, based on total count rate measurement, versus the reported isotope mass, based on spectrometric analysis for primary isotope in aqueous sludge, Am-241. The data show a linear correlation for the primary radionuclide. The correlation between estimated and reported mass is less significant for an isotope that does not significantly contribute to the overall gamma response. As a result, one can expect that there will be a high uncertainty on the secondary isotopes' estimated masses, which are derived from total count rate.

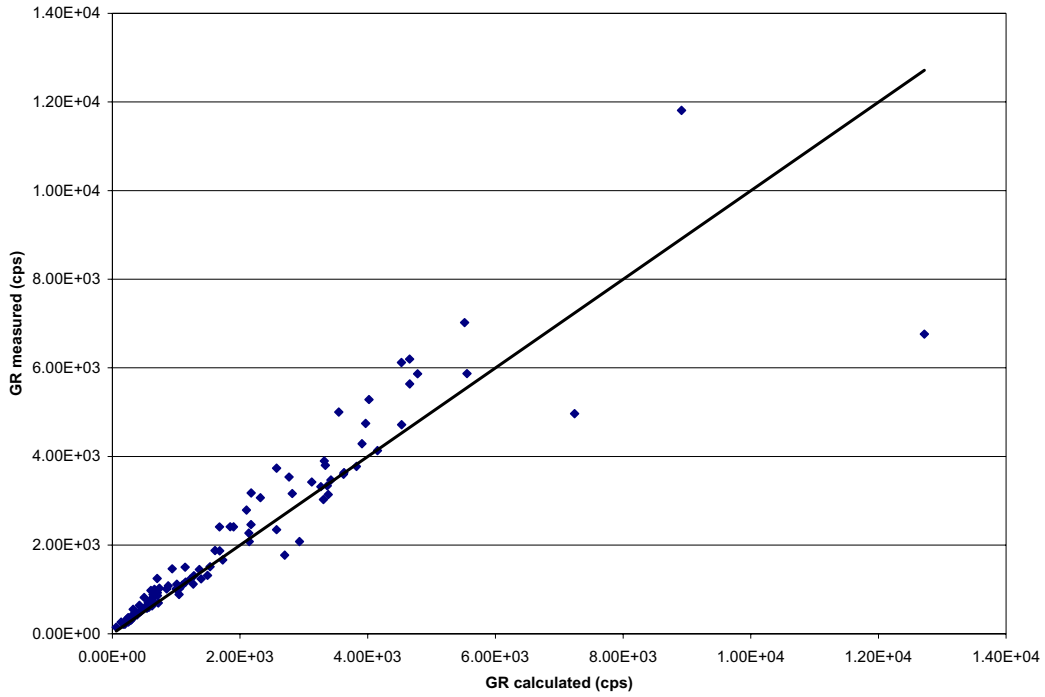

Figure 3. Americium-241 mass, as determined by gamma spectrometry, versus americium-241 mass, as determined from the total count rate method for aqueous sludge.

Derived Parameter Calculations

The derived quantities required by the Contact-Handled Waste Acceptance Criteria are total activity (Ci), alpha activity (Ci), transuranic activity (Ci), transuranic activity concentration (nCi/g), plutonium equivalent curies (Ci), fissile gram equivalent (g), thermal power (W), and thermal power density (W/ft^3). As is the case with conventional nondestructive assay systems, these derived quantities are determined from the measured isotopic mass values:

$$total\ Activity \equiv A_{tot} = \sum_i SA_i \left\langle M_i^m \right\rangle = \left\langle GA_m \right\rangle \sum_i \frac{\left\langle R_i \right\rangle}{\left(i\sum_g \right)}$$

$$alpha\ Activity \equiv A_\alpha = \sum_i br_{i\alpha}\, SA_i \left\langle M_i^m \right\rangle = \left\langle GA_m \right\rangle \sum_i \frac{br_{i\alpha} \left\langle R_i \right\rangle}{\left(i\sum_g \right)}$$

$$TRU\ Activity \equiv A_{TRU} = \sum_i br_{i\alpha} SA_i \left\langle M_i^m \right\rangle = \left\langle GA_m \right\rangle \sum_i \frac{br_{i\alpha} \left\langle R_i \right\rangle}{\left(i\sum_g \right)}$$

where $\left(i\sum_g \right) \equiv \sum_g b_{ig} \varepsilon_{tot} \left(E_{ig} \right)$

$br_{i\alpha}$ is the alpha decay branching ratio of isotope i
the summation in A_{TRU} is restricted to transuranic isotopes whose half lives are greater than 20 years.

The latter form of derived activity equations are given to show the truly independent parameters involved in determination of each derived activity. The latter forms of these equations will be useful in uncertainty propagation. The calculation to determine the transuranic concentration is shown in Equation 4, while the data determined from the TGCR method are shown in Figure 3. The correlation is linear.

Equation 4 $$A_{conc} = \frac{A_{TRU}\left(1\times10^{9}\right)}{M_{net}}$$

Where:

A_{conc} is the transuranic concentration given in units of nano-curies/gram (nCi/g)

A_{TRU} is the alpha given in units of curies

M_{net} is the net mass of waste givrn in the drum in units of gram.

The net mass of waste in the container is the gross mass of the container minus the drum mass minus the drum liner mass as shown in Equation 5

Equation 5 $$M_{net} = M_{gross} - M_{drum} - M_{liner}$$

Where:

M_{net} is the mass of the waste in the drum given in units of grams

M_{gross} is the total mass of the drum, plus the liner and the waste

M_{liner} is the mass of the drum liner.

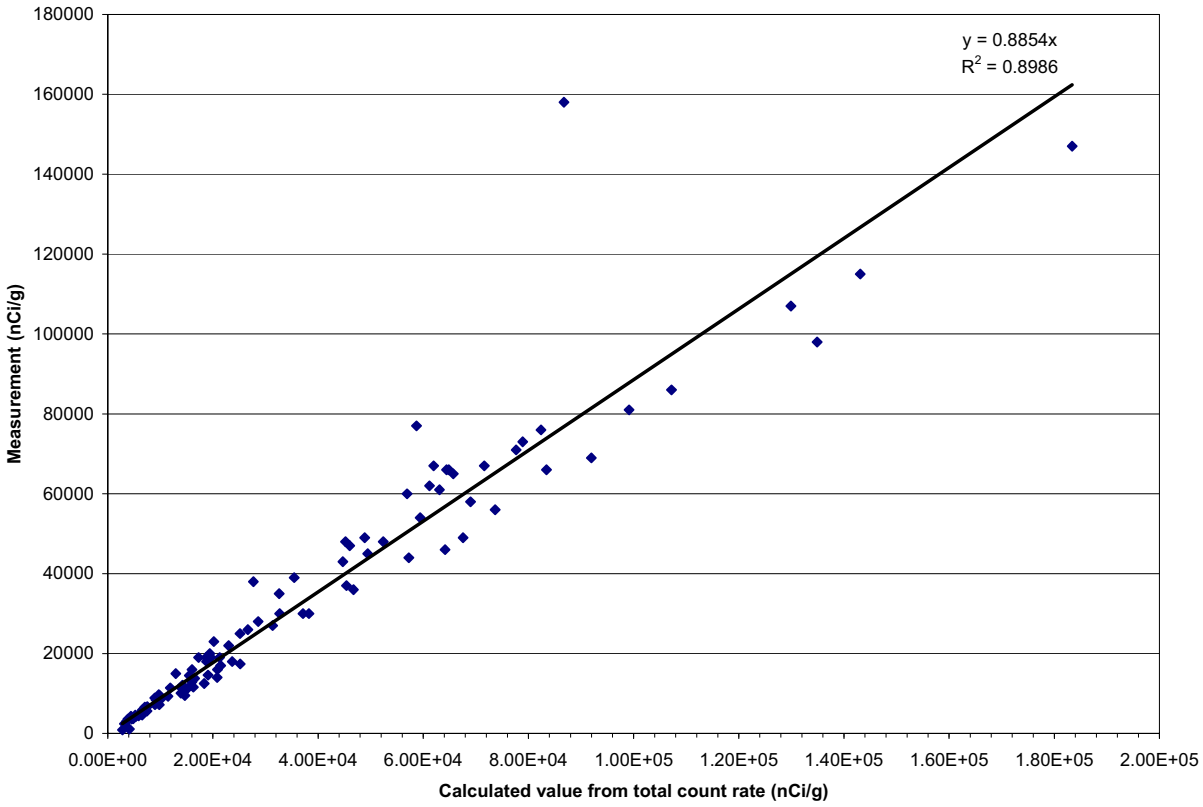

Figure 4. Transuranic concentration, as determined by gamma spectrometry, versus transuranic concentration, as determined by total gamma count rate method for aqueous sludge.

Fissile gram equivalent, as shown in Equation 6, is the equivalent plutonium mass, in units of grams, of an aggregate of isotopes based on their ability to fission in a thermal neutron field. The calculation of fissile gram equivalent follows the same prescription as the activities above, except, in the case of fissile gram equivalent, each isotope's fissile gram equivalent, F_i, divided by its specific activity, SA_i, is included in the summation over the isotopes.

Equation 6

$$FGE = \sum_i F_i \left\langle M_i^m \right\rangle = \left\langle GA_m \right\rangle \sum_i \frac{F_i \left\langle R_i \right\rangle}{SA_i \left(i \sum_g \right)}$$

Where:

FGE is the fissile gram equivalent factor given in units of grams.

F_i is the fissile gram equivalent factor for the isotope "i".

Thermal power (ThP) is, as indicated, the amount of thermal power created by the waste in units of Watts and is shown in Equation 7. The calculation of thermal power follows the same prescription as the total activity equation above, except, in the case of thermal power, the thermal power factor, Th_i, is used instead of the specific activity.

Equation 7

$$ThP = \sum_i Th_i \left\langle M_i^m \right\rangle = \left\langle GA_m \right\rangle \sum_i \frac{Th_i \left\langle R_i \right\rangle}{SA_i \left(i \sum_g \right)}$$

Where:

ThP is the derived thermal power in units of Watts (W).

Th_i is the thermal power factor for the isotope *i* in units of Watts per gram isotope (W/g).

Thermal power density is simply the thermal power divided by the volume of the waste container.

Measurements

The second phase of work performed consisted of acquiring measurements using surrogate drums. The measurements were performed in a laboratory setting, where the actual measurement conditions were simulated as much as possible. A total of six surrogate matrices will be used to perform a minimum of five sets of measurements. The following hardware/software were used to collect the data:

A single high purity germanium detector
Annular lead collar covering the sides of the detector
0.020-in. thick cadmium covering the front and sides of the detector
Canberra DSA1000
Canberra GENIE2K
Turntable
Lead containing shields on three sides of the drum and on the concrete floor.

The following sources were used:

A 10 uCi Co60
A 10 uCi Cs-137
A 10 uCi Ba-133
A 10 uCi Eu-152
A 10 uCi Na-22
A 10 uCi Mn-54
A 10 uCi Am-241
A 10 uCi Cd-109
Three 1-g plutonium foils (originally used in Nuclear Accident Dosimeters).

The measurements were performed with the following (surrogate) waste containers:

Empty drum
Graphite drum—density of 0.86 g/cm^3
Sand drum to simulate dirt—density of 1.16 g/cm^3
Aqueous sludge drum— density of 1.2 g/cm^3
Filter drum— density of 0.3 g/cm^3
Organic sludge drum— density of 1.2 g/cm^3.

The measurements performed to date clearly demonstrated that, on an average, the TGCR method obtained a minimum detection limit that was a factor of 4 lower that the photopeak analysis method (see Table 4). The worst-case graphite scenario for determining a minimum detection limit was when sources were placed at the top and bottom of the waste container. The L_d was 872 counts while, the minimum detection limit was 10.7 nCi/g for TGCR. The remainder of the measurements obtained a minimum detection limit of less than 10 nCi/g.

Table 4. Minimum detection limits in a graphite surrogate waste container for total gamma count rate.

Filename	Tube[a]	Position[b]	Tube[a]	Position[b]	Tube[a]	Position[a]	Integral (cts/uCi)	TGCR MDA (uCi)	MDL (nCi/g)	413 keV MDL (nCi/g)
NAD_09120501	3	5	3	7	3	6	9.8	89	0.53	8.6
NAD_09120502	3	5	3	7	3	6	10	87	0.52	8.0
NAD_09120503	3	5	3	7	3	6	9.6	91	0.55	7.9
NAD_09120504	3	7	2	2	2	1	3.0	287	1.7	15

NAD_09120505	3	7	2	2	2	1	3.0	286	1.7	15
NAD_09120506	3	7	2	2	2	1	3.0	290	1.7	14
NAD_09120507	2	7	3	10	3	4	2.8	312	1.9	16
NAD_09120508	2	7	3	10	3	4	2.7	321	1.9	14
NAD_09120509	2	7	3	10	3	4	2.7	318	1.9	16
NAD_09120510	3	7	2	1	3	2	3.1	284	1.7	13
NAD_09120511	3	7	2	1	3	2	3.0	290	1.7	13
NAD_09120512	3	7	2	1	3	2	3.1	281	1.7	13
NAD_09130501	2	2	3	8	3	7	3.5	246	1.5	11
NAD_09130502	2	2	3	8	3	7	3.6	241	1.4	12
NAD_09130503	2	2	3	8	3	7	3.6	242	1.5	14
NAD_09140501	3	8	3	5	2	3	4.8	183	1.1	12
NAD_09140502	3	8	3	5	2	3	4.8	182	1.1	13
NAD_09140503	3	8	3	5	2	3	4.9	177	1.1	13
NAD_09140504	3	10	3	9	3	1	0.49	1796	11	50
NAD_09140505	3	10	3	9	3	1	0.50	1729	10	51
NAD_09140506	3	10	3	9	3	1	0.48	1821	11	60
NAD_09140507	2	6	3	9	3	4	3.2	271	1.6	16
NAD_09140508	2	6	3	9	3	4	3.2	268	1.6	12
NAD_09140509	2	6	3	9	3	4	3.2	270	1.6	15
NAD_09140510	2	9	3	5	2	1	3.9	225	1.4	12
NAD_09140511	2	9	3	5	2	1	3.9	224	1.3	14
NAD_09140512	2	9	3	5	2	1	3.9	226	1.4	12
NAD_09150501	3	2	2	3	3	6	5.6	157	0.94	12
NAD_09150502	3	2	2	3	3	6	5.5	158	0.95	10
NAD_09150503	3	2	2	3	3	6	5.6	156	0.94	10
NAD_09150504	3	8	2	9	2	4	2.9	296	1.8	14
NAD_09150505	3	8	2	9	2	4	3.1	286	1.7	13
NAD_09150506	3	8	2	9	2	4	2.9	298	1.8	12
NAD_09150507	2	9	3	9	3	8	1.4	612	3.7	22
NAD_09150508	2	9	3	9	3	8	1.4	606	3.6	23
NAD_09150509	2	9	3	9	3	8	1.4	604	3.6	23

a. In the surrogate waste matrix drum, there were three guide tubes at different radial positions.
b. In the surrogate waste matrix drum, each guide tube contained a wand with eleven source positions.

MDA = minimum detectable activity
MDL = minimum detection limit
TGCR = total gamma count rate

Conclusion

Because TGCR involves all photon interactions plus multiple scattering interactions in the matrix and in the gamma detector, the TGCR method of analysis has the potential to extend the range of gamma-based assay systems by lowering the minimum detection limit. The results shown in this document indicate that the derived quantities (e.g., total activity, transuranic activity concentration, and fissile gram equivalent) can be determined by the TGCR method with reasonable accuracy as long as waste process knowledge is known. This is true even though the total count rate derived mass values for the secondary contributing isotopes have large uncertainties. Work will continue to baseline this parametric study against a model and measurements. The objective will be to establish total measurement uncertainty of TGCR method and to establish minimum detection limits. These results will be compared with corresponding results from gamma spectrometry.

It is expected that comparison data will show that the TGCR method can be used to analyze gamma spectrometer data where no measured activities can be determined by normal gamma spectrometric analysis. If this method can be

implemented at the INL Site, there is the potential of dramatically increasing the number of drums that can be sent to a proper permanent disposal site.

Reference

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2) EDF-1609, 2000, "Plutonium Mass Fractions Derived from SGRS Data," Rev. 0, Idaho National Engineering and Environmental Laboratory.